U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER	
785.40641X00 filed September 17,2001	
U.S. APPLICATION NO. (If known, see 37 CFR 1.5	
09/936696	
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/02069	09 March 2000 (9.03.00)	17 March 1999 (17.03.99)

TITLE OF INVENTION METHOD OF TREATING AND PROCESSING ALKALOID-, OIL- AND PROTEIN-CONTAINING LUPINE SEEDS

APPLICANT(S) FOR DO/EO/US ; LUCK, Thomas; BORCHERDING, Axel; HOLLEY, Wolfgang; WASCHE, Andreas; KAMAL, Hisham and MULLER, Klaus

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. [X] This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. [ ] This is a **SECOND or SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. [ ] This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. [X] The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. [X] A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. [ ] is attached hereto (required only if not communicated by the International Bureau).
   b. [X] has been communicated by the International Bureau.
   c. [ ] is not required, as the application was filed in the United States Receiving Office (RO/US).
6. [X] An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
   a. [X] is attached hereto.
   b. [ ] has been previously submitted under 35 U.S.C. 154(d)(4).
7. [ ] Amendments to the claims of the International Aplication under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. [ ] are attached hereto (required only if not communicated by the International Bureau).
   b. [ ] have been communicated by the International Bureau.
   c. [ ] have not been made; however, the time limit for making such amendments has NOT expired.
   d. [ ] have not been made and will not be made.
8. [ ] An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. [ ] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. [ ] An English lanugage translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. [ ] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. [ ] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. [X] A FIRST preliminary amendment.
14. [ ] A SECOND or SUBSEQUENT preliminary amendment.
15. [ ] A substitute specification.
16. [X] A change of power of attorney and/or address letter.
17. [ ] A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. [ ] A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. [ ] A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. [X] Other items or information:

International Publication No. WO 00/54608 (cover sheet)
International Preliminary Examination Report
International Search Report
Credit Card Payment Form
Figs. 1 & 2

531 Rec'd PCT/ 17 SEP 2001

U.S. APPLICATION NO. (If known, see 37 CFR 1.5) 09/936896	INTERNATIONAL APPLICATION NO. PCT/EP00/02069	ATTORNEY'S DOCKET NUMBER 785.40641X00

21. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a (2)) paid to USPTO and International Search Report not prepared by the EPO or JPO **$1000.00**

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO **$860.00**

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO **$710.00**

International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) **$690.00**

International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) **$100.00**

	CALCULATIONS PTO USE ONLY	
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 860.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$ 0.00	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	$	
Total claims	51 -20 =	31	**x $18.00**	$ 558.00	
Independent claims	2 -3 =	0	**x $80.00**	$ 0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			**+ $270.00**	$ 0.00	
TOTAL OF ABOVE CALCULATIONS =				$ 1,418.00	
☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2. +				$ 0.00	
SUBTOTAL =				$ 1,418.00	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				$ 0.00	
TOTAL NATIONAL FEE =				$ 1,418.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property +				$ 0.00	
TOTAL FEES ENCLOSED =				$ 1,418.00	
				Amount to be refunded:	$
				charged:	$

a. ☒ A check in the amount of $ 1,418.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of $ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 01-2135. A duplicate copy of this sheet is enclosed.

d. ☒ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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NAME

26,422
REGISTRATION NUMBER

785.40641X00

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s): H. KIMAL ET AL.

Serial No.: New Application

Filed: Herewith

For: METHOD OF TREATING AND PROCESSING ALKALOID-, OIL- AND PROTEIN-CONTAINING LUPINE SEEDS

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

September 17, 2001

SIR:

Prior to examination, please amend the above-identified application as indicated below and consider the remarks which follow:

IN THE CLAIMS:

Please cancel claims 1-38 without prejudice or disclaimer and add the following new claims:

39. A method of treating and processing alkaloid-, oil- and protein-containing lupine seeds for the extraction of products from the lupine seeds by targeted fractionation, whereby the comminuted lupine seeds are de-oiled by introducing a solvent and the residue is depleted of substances soluble in an acid

range, by adding acids, where the lupine seeds are comminuted and/or shaped to form discoid flakes so that after pre-crushing of the shelled or non-shelled seeds, the comminution of the seeds is carried out by a cooled flocculating roller, and that the seeds are heated by an indirect supply of heat substantially with exclusion of water, and that after de-oiling the depletion of the flakes of substances soluble in the acid range, is performed by aqueous extraction, with a refined product of a reduced alkaloid level and an aqueous extract being obtained.

40. A method according to claim 39,

wherein after pre-crushing of the shelled or non-shelled seeds, the comminution of the seeds is carried out by means of a flocculating roller, with the flocculating roller being cooled.

41. A method according to claim 39,

wherein the seeds are screened by shape and size prior to comminution and/or shaping and are subsequently shelled.

42. A method according to claim 39,

wherein the shelling is carried out with a technique wherein the lupine

seeds are halved and separated from the shells.

43. A method according to claim 40,

wherein the flocculating roller is cooled to a temperature lower than the denaturation temperature of the lupine proteins.

44. A method according to claim 39,

wherein the discoid flakes have a platelet thickness of less than 1 mm.

45. A method according to claim 39,

wherein the indirect heat supply is carried out by a heat pan.

46. A method according to claim 39,

wherein the indirect heat supply deactivates seed-inherent enzymes, while proteins therein substantially retaining native properties.

47. A method according to claim 39,

wherein ethanol is used as solvent in de-oiling.

48. A method according to claim 39,

wherein one of industrial hexane, pentane, hexane, heptane or supercritical CO_2 is used as a solvent for de-oiling the discoid flakes.

49. A method according to claim 47,

wherein the de-oiling process is combined with a mechanical oil separation process with the mechanical oil separation process using ethanol/water mixtures in combination with centrifuging techniques.

50. A method according to claim 39,

wherein the de-oiled discoid flakes are de-solventised.

51. A method according to claim 50,

wherein the de-solventising is carried out under substantially water-free conditions.

52. A method according to claim 50,

wherein the de-solventising is carried out with a superheated solvent.

53. A method according to claim 39,

wherein the indirect heat supply to de-oiled flakes is carried out with a

heat pan.

54. A method according to claim 50,

wherein an oil percentage in de-oiled and de-solventised flakes, relative to the percentage of dry solids, is lower than 2%.

55. A method according to claim 50,

wherein the de-oiled and de-solventised flakes are passed on to a disembitterment process including:

in a first step, the flakes are supplied into an aqueous acid medium for isolation of substances soluble in the acid medium for obtaining an aqueous acid extract as a refined product insoluble in the acid range, and

in a second step, the refined product which is insoluble in the acid range is supplied into an aqueous alkaline medium for obtaining aqueous extracts and alkaline refined products insoluble in an acid range.

56. A method according to claim 50,

wherein shells are added to de-oiled and de-solventised flakes, which are passed on, together with the flakes, to a disembitterment process including:

in a first step, the flakes with the shells are supplied into an aqueous acid

medium for isolation of substances soluble in the acid medium to provide an aqueous acid extract and a refined product insoluble in the acid range, and

in a second step, the refined product which is insoluble in the acid range is supplied into an aqueous alkaline medium for obtaining aqueous extracts and alkaline refined products insoluble in an acid range.

57. A method according to claim 56,

wherein prior to the addition to the flakes, the shells are ground.

58. A method according to claim 55,

wherein the aqueous acid medium in the first process step has a temperature lower than room temperature.

59. A method according to claim 55,

wherein isolation of the aqueous acid extract from the refined product insoluble in the acid range is carried out centrifugally by a decanter, and the decanter is cooled and flushed in water or an extract in a zone of a solids accumulator.

60. A method according to claim 55,

wherein in the second process step a temperature for extraction in the aqueous alkaline medium is higher than the room temperature.

61. A method according to claim 55,

wherein the first process step is in a multi-stage aqueous acid process, and further comprising a process step for adjustment of a ratio between the refined product insoluble in the acid range and the aqueous extract to less than 10:1, one part of the aqueous extract from an immediately preceding process step is admixed.

62. A method according to claim 55,

further comprising a process step for adjustment of a ratio between the refined product insoluble in the acid range and the aqueous extract of more than 10:1, an outward transfer of one part of the aqueous extract is carried out within an immediately preceding process step.

63. A method according to claim 55, further comprising a process step for obtaining a product from the aqueous acid extract an isolation of substances is carried out by a separator so that a product is obtained having a concentration of dry solids at least 10%, a protein concentration in the dry solids higher than

70%, and an alkaloid level lower than 0.5%.

64. A method according to claim 63,

wherein in isolation of the substances by means of a separator is carried out in the first process step using aqueous acid process steps, and the isolation of the substances is carried out after one of the first process step or a preceding process step.

65. A method according to claim 55,

wherein the aqueous extraction includes a closed circuit providing the following process stages:

the de-oiled flakes are suspended in water at a pH level of substantially between 3.5 to 5.5 for separation of substances soluble in the acid range,

for protein extraction, suspended flakes are mixed with lye at a pH level between 7.0 and 8.5,

suspension is separated, by a decanter, to obtain a refined product and the protein extract,

an acid medium is supplied to the protein extract again to achieve fractioning of the whey and protein curds, and

the whey is supplied again completely to the pre-extracted flakes at a pH

level of substantially between 3.5 to 5.5.

66. A method according to claim 65,

wherein protein extraction is carried out in pH level stages for achieving protein fractioning.

67. A method according to claim 65,

wherein the refined product has a protein concentration less than 20% in the dry solids, a roughage percentage is higher than 60%, and a percentage of soluble carbohydrates is lower than 5%.

68. A method according to claim 65,

wherein isolation of whey and protein curds containing more than 85% of proteins in the dry solids, is carried out by a decanter.

69. A method according to claim 68,

wherein extracted whey is subjected to a first purification by means of a separator, then to a thermal treatment, and finally a second purification in a separator.

70. A method according to claim 69,

wherein twice purified whey is supplied into a process again, wherein the solids obtained in a first separation are subjected to further processing in a protein leg and with outward transfer of the solids obtained in another separation.

71. A method according to claim 65,

wherein the refined product is fractioned by particle sizes into at least 2 fractions after or during a drying stage.

72. A method according to claim 65,

wherein after drying, pressed protein curds have a protein dispersibility index (PDI) of 60 to 90% and a water-absorption capacity of less than 2 g/g at a pH level of about 7 and a temperature of 20 to 30°C.

73. A method according to claim 65,

wherein the protein curds are confectioned by a hydro-thermal treatment to form a water binding product, with application of a temperature higher than 65°C, for drying the protein curds and with a water percentage at a beginning of drying of less than 85%, while a water absorption capacity of the water binding product is higher than 4.0 g/g.

74. A method according to claim 39,

wherein mixtures of obtained roughage and the protein isolates are produced, having protein level ranges between 20 and 70%, roughage concentration ranges between 30 and 80%, and a water absorption capacity is higher than 5 g/g.

75. A method according to claim 39,

wherein shells separated prior to the de-oiling are mixed and dried with the aqueous extract at pH levels from 3.5 to 5.5

76. A method of treating and processing alkaloid-, oil- and protein-containing seeds for the extraction of products from the seeds by targeted fractionation, whereby the comminuted seeds are de-oiled by introducing a solvent and the residue is depleted of substances soluble in an acid range, by adding acids, where the seeds are comminuted and/or shaped to form discoid flakes so that after pre-crushing of the shelled or non-shelled seeds, the comminution of the seeds is carried out by a cooled flocculating roller, and that the seeds are heated by an indirect supply of heat substantially with exclusion of water, and that after de-oiling the depletion of the flakes of substances soluble in

the acid range, is performed by aqueous extraction, with a refined product of a reduced alkaloid level and an aqueous extract being obtained.

77. A method in accordance with claim 76,

wherein the seeds are selected from the group consisting of rape, linseed, soybeans, peanuts, peas and horse peas.

78. A method according to claim 42, wherein the denaturation temperature is lower than 35°C.

79. A method according to claim 46, wherein platelet thickness ranges between 200 and 400 μm.

80. A method according to claim 52, wherein the superheated solvent is hexane.

81. A method according to claim 54, wherein the oil percentage is less than 1%.

82. A method according to claim 57, wherein the particle size is less

than 5 mm.

83. A method according to claim 60, wherein the temperature for extraction ranges between 35°C and 45°C.

84. A process according to claim 63, wherein the concentration of dry solids is higher than 16%, the protein concentrates in the dry solids is higher than 85% and an alkaloid level is less than 0.1% in the dry solids.

85. A process according to claim 67, wherein the roughage concentration is higher than 70% and the percentage of soluble carbohydrates is lower than 1%.

86. A method according to claim 68, wherein the whey and protein curds contain more than 90% proteins in dry solids.

87. A method according to claim 71, wherein the refined product is refined by particle sizes into at least three fractions after or during a drying step.

88. A method according to claim 73, wherein the temperature is higher

than 85°C, a water percentage at the beginning of the drying step is less than 75% and the water absorption capacity of the water binding product is higher than 5 g/g.

89. A method according to claim 74, wherein the water absorption capacity is higher than 7 g/g.

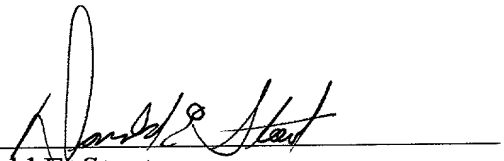
REMARKS

The claims have been amended to improve their form for examination and further to remove multiple dependent claims for fee calculation purposes.

To the extent necessary, applicants petition for an extension of time under 37 CFR §1.136. Please charge any shortage in the fees due in connection with the filing of this paper, including extension of time fees, to Deposit Account No. 01-2135 (785.40641X00) and please credit any excess fees to such deposit account.

Respectfully submitted,

ANTONELLI, TERRY, STOUT & KRAUS, LLP



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Method of Treating and Processing Alkaloid-, Oil- and Protein-Containing Lupine Seeds

Field of the invention

The invention relates to a method of treating and processing alkaloid-, oil- and protein-containing lupine seeds for the extraction of products from the lupine seeds by means of targeted fractionation, whereby the comminuted lupine seed is de-oiled by introducing a solvent and the residue is depleted of substances soluble in the acid range, preferably of alkaloids, by adding acids.

Prior art

Proteins or protein preparations, respectively, are considered to be raw materials for the food-processing and fodder industries and are used in manifold applications in industrial chemistry, for instance for the production of adhesives, emulsions for photographic layers or cosmetics, just to name some of them.

As proteins are an essential component in animals and plants they are renewable native raw materials suitable for extraction from milk, soybeans and wheat on an industrial scale, for instance. Lupine seeds, which are similar to soybeans in terms of their composition in view of protein level, crude fibre fraction and oil concentration, are particularly important. Lupine cultivation and the processing of lupine seeds for the extraction of desired protein products is therefore of particular interest because lupines can be grown also in regions unsuitable for soy beans, for example in Western Europe or Australia.

Due to plant-inherent bitter principles, the so-called alkaloids, a direct utilisation of lupine products is limited, particularly for food applications, and in the case of so-called bitter lupines, which are expedient in terms of cultivation, it is even entirely precluded. When lupine seeds are processed it is therefore necessary to remove the alkaloids in order to obtain

products for nutritional use. At the same time, the extracted alkaloids may be selectively employed as active ingredients in agriculture and in the pharmaceutical industry, which renders the complete utilisation of lupines or bitter lupines, respectively, extremely interesting from an economic point of view as well.

The German Patent DE 537 265, published in 1931, disclosed, for instance, a method of useful utilisation of lupines with disembitterment by stepwise extraction with aqueous solutions. Disembitterment is carried out by stepwise extraction of chipped lupines in moist state, with the addition of an acid and subsequent dissolution of the salts forming in the acid bath.

Moreover, the document WO 83/00419 discloses a method of and a device for extraction of the bitter ingredients from the seeds of bitter lupines after cold washing of the lupines, which are present in an extremely finely ground condition, with lupine extract solutions of different concentrations, on the counter-flow principle, with water being used as solvent.

One improved method of disembittering lupine seeds is disclosed in the document WO 97/12524 that provides for an initial thermal treatment of the plant seeds, after comminution of the lupine seeds to grit-like grains having diameters between 200 and 600 µm, so as to achieve a selective deactivation of enzymes present in the plant seeds. The thermal treatment is performed directly by means of a blanching technique, i.e. by direct introduction of hot steam into the comminuted seeds. After the blanching step, the plant seeds are subjected to a two-stage process for disembitterment, wherein the first extraction step results in the extraction of the alkaloids as well as other anti-nutritive substances. To this end, the plant seeds are mixed with fresh potable water as solvent in an acid medium in a counter-flow extraction operation. The mixing operation may be preferably carried out in several stages until an extract enriched with anti-nutritive substances and an extractable refined product are obtained, that is rich in proteins and roughage. The refined product obtained from the first extraction step is added with water as solvent in an alkaline medium in a second step. A refined product enriched with roughage as well as a protein milk enriched with proteins are obtained as results of the second extraction step.

All the aforedescribed disembitterment methods are based on a common objective, specifically the objective of extracting, on the one hand, proteins in the purest form possible and, on the other hand, of obtaining roughage for the food or fodder industries, which are disembittered as completely as possible.

The aforedescribed methods, however, present various inherent disadvantages, too: Firstly, plant seeds and lupine seeds in particular have an oil level of roughly 10 to 15 %, including lipophilic secondary ingredients such as carotinoids, lecithins or lipophilic alkaloids, in addition to pure oil such as triglycerin.

Even though the known method according to the document WO 97/12524 proposes a deactivation of the enzymes present in the plant seeds, which precedes the disembitterment process, so that the situation may be precluded that an enzymatic oxidation of existing unsaturated fatty acids will occur during storage of the disembittered products of this process, which could result in a rancid flavour, for instance, which were inexpedient for application in the food sector, the deactivation is carried out by means of blanching, which means that the plant seeds are exposed to hot steam, which, even though it deactivates the enzymes, on the one hand, creates, on the other hand, unavoidable damage to the storage proteins as well so that they lose their native form and characteristics.

Finally, the shaping of the comminuted lupine seeds contributes to the success of the disembitterment process, too. The form of grit grains proposed in the document WO 97/12524 is thus inexpedient insofar as it encloses a comparatively large volume from which the individual components to be extracted must be removed, which means that as the spacing between the interior of the volume and the outside of each grit particle increases, the substances to be extracted are less easily extracted from the grit-shaped lupine seed fragments to be disembittered. On the other hand, the document WO 83/00419 proposes the grinding of the lupine seeds to be disembittered to produce an extremely fine meal with grain sizes between 1 µm and 50 µm; this fine grinding of the lupine seeds to form a meal, however, problems occur in terms of process engineering in the separation of the liquid from the solid phase - even though the individual extraction paths inside a "dust grain" are kept very small. This requires complex filtering steps expensive in terms of process engineering, which involve a substantial cost and time factor in application on an industrial scale.

Another known disembitterment process is described in the laid-open German Patent Application DE-OS 29 08 320, wherein the lupine seeds are comminuted and de-oiled. The protein-containing residue produced in that process is subsequently heated and extracted with the addition of an acid. For this known method, too, the aforedescribed disadvantages such as the introduction of water for enzyme deactivation or insufficient comminution of the lupine seeds must be mentioned.

It is also possible to use any protein- and oil- or starch-containing seed, in addition to the aforementioned lupine seed, such as rape, linseed or leguminous plants, particularly soybeans, peanuts, peas and horse beans.

Brief description of the invention

The present invention is based on the problem of proposing a method of treating and processing alkaloid-, oil- and protein-containing lupine seeds for the production of products from lupine seeds by way of targeted fractioning in such a way that the products - proteins in extremely pure form as well as roughage - can be freed of bitter principles as completely as possible, wherein the steps of operation to be carried out in succession should involve the smallest technological expenditure possible. On the one hand, particular attention must be paid to the aspect that the proteins to be treated should remain unchanged in their native form whilst enzymes present in the lupine seeds should be deactivated and lipophilic alkaloids, in particular, should be extracted as completely as possible in the most gentle manner. The method is intended to improve the degree of disembitterment of lupine seeds, that has so far been achieved, substantially by the simplest process steps possible, which are harmonised with each other, or to reduce, respectively, the engineering expenditure substantially with the same disembitterment result.

In accordance with the present invention, the following steps of operation are carried out in the method of treating and processing alkaloid-, oil- and protein-containing lupine seeds for the extraction of products from the lupine seeds that may be both rich in bitter principles - the so-called bitter lupines - or poor in bitter principles, by way of targeted fractioning:

First of all, the lupine seeds are shelled and the shells are isolated. Then the kernel meat of the seeds is comminuted or shaped, respectively, to produce discoid flakes, e.g. by passing them through a flocculating roller. The flocculating roller is cooled so that the comminution process becomes more efficient and gentler for the seeds to be comminuted. Cooling is particularly intended to prevent the seeds from heating during comminution. The cooling effect can be ensured, for example, with common tap water that maintains the comminuted seeds in a temperature range still below the denaturation temperature of the lupine proteins. Suitable temperatures are within the range between 8 °C and 35 °C. Then heat is indirectly supplied into the flakes, with water being largely excluded. Such indirect heating is carried out in a heat pan into which the comminuted flakes are passed. Due to the gentle indirect supply of heat the enzymes contained in the lupine seeds are deactivated while the proteins remain largely in their original form and retain their functional properties in unmodified form because

they do not come into direct contact with water, which would cause damage to the natural properties of the proteins.

Then the flakes are selectively subjected to a de-oiling process in which a solvent, preferably hexane, is used that permits the extraction of the lipids contained in the discoid flakes. It is equally possible to use alternatively ethanol, industrial hexane, pentane, heptane or supercritical CO_2 instead of hexane. Moreover, the de-oiling process with the aforementioned solvent may also be combined with a mechanical oil separation in the form of presses or with a de-oiling step using ethanol/water mixtures and applying centrifuging techniques.

The extracted lipids concern, in particular, also all the lipophilic alkaloids contained in the lupine seeds, which may be isolated by a de-oiling step so that merely lipophobic alkaloids are present as bitter principles in the hexane-wet discoid flakes, which must then be extracted in a subsequent disembitterment process. The flakes de-oiled and de-solventised in the afore-described manner present preferably an oil concentration of less than 2 %, preferably of less than 1 %, of the dry solid. The solvent is preferably removed without water in an overheated solvent such as hexane. On principle, however, any other optional de-solventising method is applicable. The benzene is preferably extracted from the hexane-wet meal in a gentle manner, e.g. using supercritical hexane.

For disembitterment the discoid flakes, from which benzene is removed and in which the lipid level is reduced, are subjected to an aqueous fractioning process. It is also possible to mix shell fractions to the flakes of a reduced lipid level, which had been reduced to a granularity of less than 5 mm in a preceding grinding step. The disembitterment process substantially comprises two stages:

Initially, the de-oiled flakes - possible together with shell fractions - are introduced into an aqueous acid medium in which all those substances dissolve which are contained in the flakes and which are soluble in the acid range. As a result, one obtains an aqueous acid extract containing the alkaloids in particular, as well as a disembittered refined product insoluble in the acid range, that consists substantially of the flake substance.

The flakes so extracted - which are also referred to as meal - may be subjected to a further subsequent extraction aiming at the production of isolated protein products or protein concentrates. The subsequent extraction involves aqueous systems, too, which may be provided in several stages in succession. The solid phase can be isolated from the liquid phase by means of decanting for obtaining the protein extract as well as products or compartments

depleted of proteins, while it is possible to control the protein level remaining in the residual flaky substance by defined process conditions such as the pH level, the extraction times as well as temperatures.

It is furthermore possible to obtain a product from the aqueous acid extract, which can be produced by an isolation of fine particles by means of a separator. As the first step of the method is a multi-stage process in some kind of cascade including a plurality of aqueous acid process steps inn succession the fine substance is isolated after the passage through the first process stage at the earliest. In one process stage, that serves to adjust a ratio of less than 10:1 between the refined products insoluble in the acid range and the aqueous extract, one part of the aqueous extract is added to the immediately joining process stage. It is also possible to set a ratio of more than 10:1 between the refined products insoluble in the acid range and the aqueous extract by outward transfer of one part of the aqueous extract.

The product that can be obtained in this step hence presents a level of dry solids of at least 12 %, preferably higher than 16 %, a protein level in the dry solids of more than 70 %, preferably higher than 85 %, and an alkaloid percentage of less than 0.5 %, preferably 0.1 % in the dry solids. Moreover, the product contains roughage that is fractioned by particle sizes into at least 2, preferably 3, fractions after or during the drying phase.

When the refined product insoluble in the acid range, which is obtained after the first process stage, is introduced into an aqueous alkaline medium in which all those substances are dissolved which dissolve in the alkaline range, i.e. at pH levels of more than 7.5, a refined product of reduced alkaloid level is obtained as final result immediately after the second process step, which is not only freed of any lipophilic alkaloids but also of alkaloids soluble in the acid range.

The refined product of reduced alkaloid level, which is also referred to as protein curds, is preferably dried and presents a protein dispersibility index of 60 to 90 % and a water-absorption capacity of less than 2g/g at a pH level of roughly 7 and a temperature of 20 to 30 °C after drying.

Moreover, the protein curds may be confectioned by hydrothermal treatment to form a water binding product, with application of a temperature higher than 65 °C, preferably 85 °C, for drying the protein curds and with a water percentage at the beginning of the drying step of less than 85 %, preferably less than 75 %, while the water absorption capacity of the water binding product to be obtained is higher than 4.0 g/g, preferably higher than 5 g/g.

The aforedescribed method is suitable for application not only in the treatment of lupine seeds but also for processing other protein- and oil- or starch-containing seeds such as rape, linseed or leguminous seeds, particularly soy beans, peanuts, peas and horse beans.

Brief description of the figures

The present invention will be described in the following by exemplary embodiments, without any limitation of the general inventive idea, with reference to the drawing wherein:

Figs. 1, 2 show a schematic of the process for de-oiling and disembitterment of alkaloid-containing lupine seeds.

Brief description of an embodiment

Fig. 1 illustrates a block schematic of the first three process steps. In the first process step 100 the lupine seeds are prepared, in the second process step 200 de-oiling takes place, and in the third process step 300 disembitterment is carried out.

The starting material for the method is lupine seed that is comminuted and shelled in a preparatory step. The lupine seeds isolated in this manner are then flocculated, preferably in the course of a rolling operation, which means that the lupine seeds are pressed to form seed fragments having a typical platelet thickness between 300 and 400 µm. The flocculating roller used for the rolling operation is cooled, not least in an approach to enhance the efficiency of the comminuting operation.

After comminution, the flakes arrive in a heat pan where they are subjected to indirect thermal treatment. Even though this thermal introduction of heat deactivates, on the one hand, the seed-inherent enzymes the native properties of the proteins are maintained to the largest possible extent, so that later enzymatic fat oxidations, which would result in rancid flavours, may be precluded. The flocculated lupine seed, which has moreover been enzymatically deactivated, is now passed on to a subsequent de-oiling process 200 where the flakes are exposed to hexane as solvent so that any lipophilic substances such as triglycerins and crude lecithins as well as lipophilic alkaloids in particular may be extracted. This is typically performed in a belt-type or rotary extractor. The liquid phase is subjected to distillation; in this step the used solvent, hexane, is firstly recovered and made available for re-utilisation and secondly the extracted crude oil R can be purified in a further refining process that is not illustrated in the Figure. The crude lecithins can be further refined by use of acetone.

The hexane-wet de-oiled flakes present in de-oiling after the extraction process 200 are isolated from the solvent in the gentlest manner possible, which means that they are de-solventised. In this step it is particularly essential that the solubility of the proteins is either retained are can bee selectively varied as far as this is feasible from a technological point of view. To this end, the hexane-wet flakes are de-solventised, e.g. by application of an overheated solvent, under low-water conditions.

The de-oiled flake-shaped lupine seeds from this preliminary processing step are than freed of any alkaloids still present in the lupine seeds in a disembitterment step 300. The lupine seed disembitterment takes place in a multi-stage aqueous disembitterment process in a manner known per se, in which the alkaloids may be extracted continuously, quasi continuously or in batches, as is illustrated in Fig. 1.

Initially, the de-oiled flakes are passed into an acid medium where all those substances - and particularly alkaloids - are dissolved which are soluble in an acid medium. The flakes processed in this manner are then passed to the protein extraction stage 400 according to Fig. 2, where the flakes are exposed, for instance repeatedly, to an alkaline medium, with fractioning taking place between the refined product and the protein extracts. In acid media the proteins can then be precipitated from the protein extract. The whey produced during the protein precipitation, whose pH level corresponds to that of the acid medium for disembitterment of the lupine seeds in the disembitterment stage 300, may be recycled in a closed circuit into the disembitterment process 300 again.

The residue presenting a reduced protein level is then passed on as substance flow for roughage processing for obtaining a refined product in the zone of the roughage processing stage 600 where the flakes are neutralised by the addition of an appropriate acid and are then dried. On the other hand, the protein extract produced in protein precipitation may result in the protein product directly when neutralisation is appropriately carried out with the addition of alkaline media and subsequent drying. In an alternative, the functional properties of parts of the protein extract may be modified in the process step 500 by suitable thermal heat treatment or selective application of high-frequency fields, and in this manner, after drying, a refined protein product can be achieved.

It is not only possible to obtain the products of the raffinate, which corresponds to the roughage, as well as the protein products, but also to achieve bitter principle extracts selectively from the disembitterment process, which occur, for instance, as extract containing bitter principles in the bitter extract processing stage 700. To this end, bitter extracts are selectively

removed from the disembitterment process 300, which, after appropriate processing steps such as isolation of fine substances, neutralisation and evaporation to dryness, result in the final product.

It is equally possible to admix the shells separated in the process step 100 to the extract containing bitter principles. The extract so produced and fixed on shells may then be dried.

The essential aspect of the described inventive method of treating and processing alkaloid-, oil- and protein-containing lupine seeds consists in the aspect that the lipophilic alkaloids, which are very difficult to extract in the development process, have been extracted from the lupine seeds already in a preceding de-oiling process. In this manner one can largely completely preclude the existence of alkaloids in the products obtained by the end of the process. In accordance with the invention, the comminution of the lupine seeds to form flakes equally contributes to the possibility that firstly the bitter principles contained in the lupine seeds may completely escape from the seeds and secondly that the liquid and solid phases can be easily isolated in a technologically simple operation. Moreover, the behaviour of the alkaloids in aqueous systems is substantially improved by removal of the lipophilic seed ingredients. This produces a positive influence particularly on the necessary dwelling times in the various extraction stages.

List of Reference Numerals

100	preliminary treatment of the seeds, flocculation, deactivation
200	de-oiling step
300	disembitterment step
400	protein production
500	protein refining step
600	processing the refined product
700	processing the bitter principles

PATENT CLAIMS

1. Method of treating and processing alkaloid-, oil- and protein-containing lupine seeds for the extraction of products from the lupine seeds by means of targeted fractionation, whereby the comminuted lupine seed is de-oiled by introducing a solvent and the residue is depleted of substances soluble in the acid range, preferably of alkaloids, by adding acids, **characterised** in that the lupine seeds are comminuted and/or shaped to form discoid flakes in such a way that after pre-crushing of the shelled or non-shelled seed containing the plant seeds the comminution of the plant seeds is carried out by means of a cooled flocculating roller, and that the seed is heated by indirect supply of heat, largely with exclusion of water, and that after de-oiling the depletion of the flakes of substances soluble in the acid range, preferably of alkaloids, is performed by aqueous extraction, with a refined product of a reduced alkaloid level and an aqueous extract being obtained.

2. Method according to Claim 1,
characterised in that after pre-crushing of the shelled or non-shelled seed containing the plant seeds, the comminution of the plant seeds is carried out by means of a flocculating roller, with said flocculating roller being cooled.

3. Method according to Claim 1 or 2,
characterised in that the lupine seeds are screened by shape and size prior to comminution and/or shaping and are subsequently shelled.

4. Method according to any of the Claims 1 to 3,
characterised in that the shelling operation is carried out in correspondence with the so-called cold technique wherein the lupine seeds are halved and separated from the shells.

5. Method according to any of the Claims 1 to 4,
characterised in that said flocculating roller is cooled down to a temperature lower than the denaturation temperature of the lupine proteins, preferably lower than 35 °C.

6. Method according to any of the Claims 1 to 5,
characterised in that said discoid flakes present a platelet thickness of less than 1 mm, preferably in the range between 200 and 400 µm.

7. Method according to any of the Claims 1 to 6,
characterised in that the indirect heat supply is carried out by means of a heat pan.

8. Method according to any of the Claims 1 to 7,
characterised in that the indirect heat supply deactivates seed-inherent enzymes, with the proteins retaining their native properties as largely as possible.

9. Method according to any of the Claims 1 to 8,
characterised in that in the de-oiling step ethanol is used as solvent.

10. Method according to any of the Claims 1 to 8,
characterised in that industrial hexane, pentane, hexane, heptane or supercritical CO_2 is used as solvent for de-oiling the discoid flakes.

11. Method according to Claim 9 or 10,
characterised in that the de-oiling process is combined with a mechanical oil separation process with pressing or with a de-oiling process operating on ethanol/water mixtures, with application of centrifuging techniques.

12. Method according to any of the Claims 1 to 11,
characterised in that the de-oiled discoid flakes are de-solventised.

13. Method according to Claim 12,
characterised in that the de-solventising process is carried out under low-water or water-free conditions.

14. Method according to Claim 12 or 13,
characterised in that the de-solventising process is carried out with a superheated solvent that is preferably hexane or industrial hexane.

15. Method according to any of the Claims 1 to 14,
characterised in that the indirect heat supply to the flakes already de-oiled is carried out by means of a heat pan.

16. Method according to any of the Claims 12 to 15,
characterised in that the oil percentage in the de-oiled and de-solventised flakes, relative to the percentage of dry solids, is lower than 2 %, preferably lower than 1 %.

17. Method according to any of the Claims 12 to 16,
characterised in that the de-oiled and de-solventised flakes are passed on to a disembitterment process providing the following two steps of operation:

- in a first step, the flakes are supplied into an aqueous acid medium for isolation of substances soluble in the acid medium for obtaining an aqueous acid extract as well as a refined product insoluble in the acid range,
- in a second step, the refined product insoluble in the acid range is supplied into an aqueous alkaline medium for obtaining aqueous extracts as well as alkaline refined products insoluble in the acid range.

18. Method according to any of the Claims 12 to 16,
characterised in that shells are added to the de-oiled and de-solventised flakes, which are passed on, together with the flakes, to a disembitterment process providing the following two steps of operation:

- in a first step, the flakes with the shells are supplied into an aqueous acid medium for isolation of substances soluble in the acid medium for obtaining an aqueous acid extract as well as a refined product insoluble in the acid range,
- in a second step, the refined product insoluble in the acid range is supplied into an aqueous alkaline medium for obtaining aqueous extracts as well as alkaline refined products insoluble in the acid range.

19. Method according to Claim 18,
characterised in that prior to the addition to the flakes, the shells are ground, preferably to a particle size smaller than 5 mm.

20. Method according to any of the Claims 17 to 19,
characterised in that the aqueous acid medium in the first process step has a temperature lower than room temperature.

21. Method according to any of the Claims 17 to 20,
characterised in that the isolation of the aqueous extract from the refined product insoluble in the acid range is carried out centrifugally by means of a decanter, and that the decanter is cooled and flushed with water or the extract in the zone of the solids accumulator.

22. Method according to Claim 17 or 18,
characterised in that in the second process step the temperature for extraction in the aqueous alkaline medium is higher than the room temperature and preferably ranges between 35 °C and 45 °C.

23. Method according to any of the Claims 17 to 22,
characterised in that the first process step takes place in a multi-stage aqueous acid process,
that in a process step for adjustment of a ratio between the refined product insoluble in the acid range and the aqueous extract of less than 10:1, one part of the aqueous extract from the immediately joining process step is admixed.

24. Method according to Claim 25,
characterised in that for adjustment of a ratio between the refined product insoluble in the acid range and the aqueous extract of more than 10:1, an outward transfer of one part of this aqueous extract is carried out within the scope of the immediately joining process step.

25. Method according to any of the Claims 17 to 24,
characterised in that for obtaining a product from the aqueous acid extract an isolation of fine substances is carried out by means of a separator such that a product is obtained whose concentration of dry solids is at least 10 %, preferably higher than 16 %, whose protein concentration in the dry solids is higher than 70 %, preferably higher than 85 %, and whose alkaloid level is lower than 0.5 %, preferably 0.1 %, in the dry solids.

26. Method according to Claim 25,
characterised in that the isolation of fine substances by means of a separator is carried out within the scope of the first process step, that involves several aqueous acid process steps, and that the isolation of fine substances is carried out after the first process step or a joining process step.

27. Protein preparation according to any of the Claims 17 to 19,
characterised in that the process of aqueous extraction includes a closed circuit providing the following process stages:

- the de-oiled flakes are suspended in water at a pH level of roughly 3.5 to 5.5 for separation of substances soluble in the acid range, preferably of alkaloids,

- for protein extraction, the suspended flakes, the so-called protein extract, are mixed with a lye at a pH level between 7.0 and 8.5,
- the suspension is separated, by means of a decanter, to obtain a refined product and the protein extract,
- an acid medium is supplied to the protein extract again so as to achieve fractioning of whey and protein curds, and
- the whey is supplied again completely to the pre-extracted flakes at a pH level of roughly 3.5 to 5.5.

28. Method according to Claim 27,
characterised in that the protein extraction is carried out in several pH level stages for achieving protein fractioning.

29. Method according to any of the Claims 27 and 28,
characterised in that the refined product has a protein concentration of less than 20 % in the dry solids, and that the roughage percentage is higher than 60 %, preferably 70 %, and that the percentage of soluble carbohydrates is lower than 5 %, preferably lower than 1 %.

30. Method according to Claim 27,
characterised in that the isolation of whey and protein curds containing more than 85 % of proteins in the dry solids, preferably more than 90 % of proteins in the dry solids, is carried out by means of a decanter.

31. Method according to Claim 30,
characterised in that the extracted whey is subjected to subsequent purification by means of a separator, then to a thermal treatment, and finally to a second step of purification in a separator.

32. Method according to Claim 31,
characterised in that the whey purified twice is supplied into the process again, wherein the solids obtained in the first separation are subjected to further processing in the protein leg and with outward transfer of the solids obtained in the second separation.

33. Method according to Claim 27,
characterised in that the refined product is fractioned by particle sizes into at least 2, preferably 3, fractions after or during a drying stage.

34. Method according to Claim 27,
characterised in that after drying the protein curds present a protein dispersibility index (PDI) of 60 to 90 % and a water-absorption capacity of less than 2g/g at a pH level of roughly 7 and a temperature of 20 to 30 °C.

35. Method according to the Claims 27, 28, 30 and 32,
characterised in that the protein curds is confectioned by a hydro-thermal treatment to form a water binding product, with application of a temperature higher than 65 °C, preferably higher than 85 °C, for drying the protein curds and with a water percentage at the beginning of the drying step of less than 85 %, preferably less than 75 %, while the water absorption capacity of the water binding product so obtained is higher than 4.0 g/g, preferably higher than 5 g/g

36. Method according to any of the Claims 1 to 35,
characterised in that mixtures of roughage and the protein isolates obtained are produced, whose protein level ranges between 20 and 70 %, whose roughage concentration ranges between 30 and 80 %, and whose water absorption capacity is higher than 5 g/g, preferably higher than 7 g/g.

37. Method according to any of the Claims 1 to 36,
characterised in that the shells separated prior to the de-oiling step are mixed and dried with the aqueous alkaloid-containing extract that is extracted at pH levels from 3.5 to 5.5.

38. Method according to any of the Claims 1 to 37,
characterised in that other protein- and oil- or starch-containing seeds such as rape, linseed or leguminous plants, specifically soy beans, peanuts, peas and horse beans, are used instead of lupines in this method.

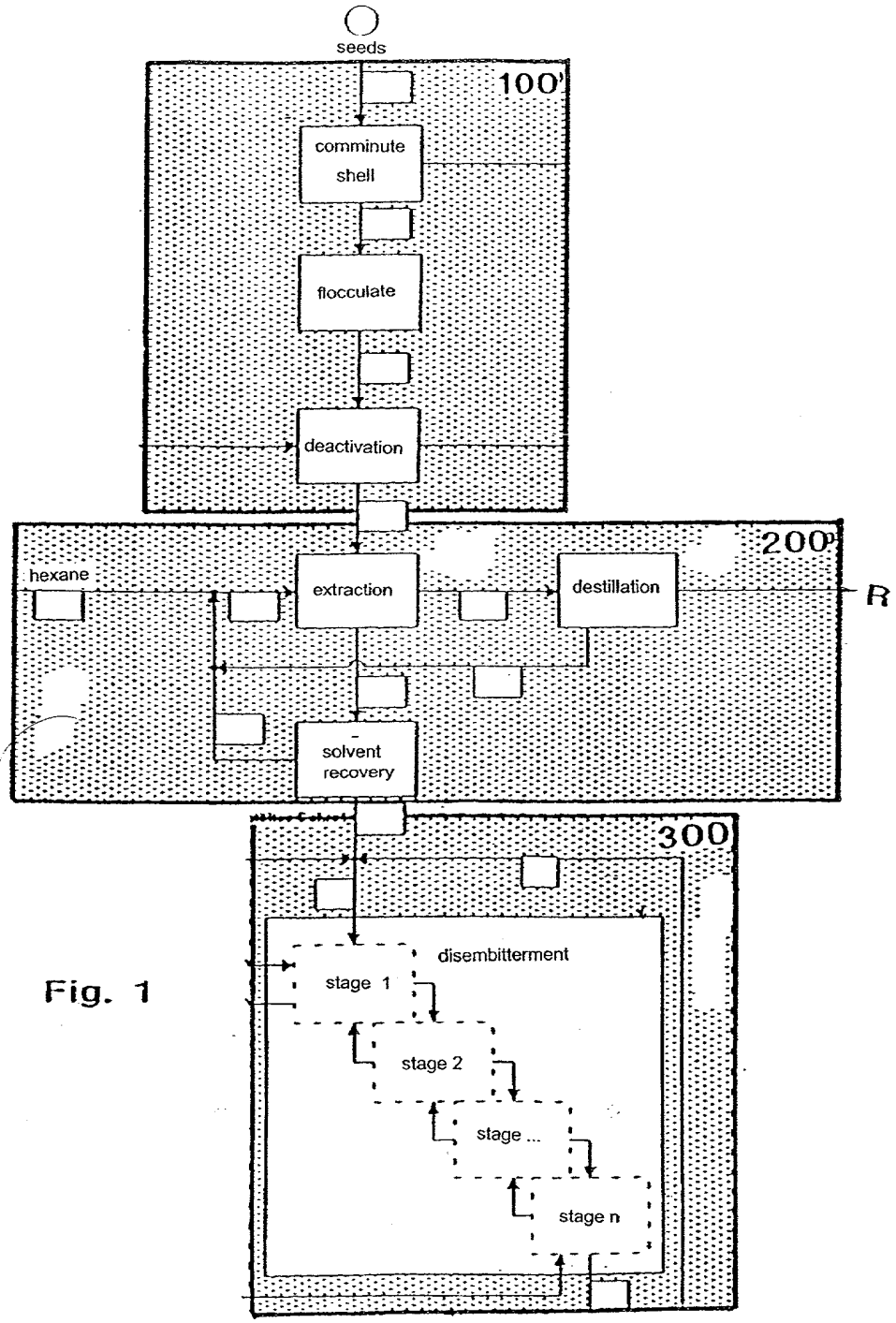
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Fig. 1

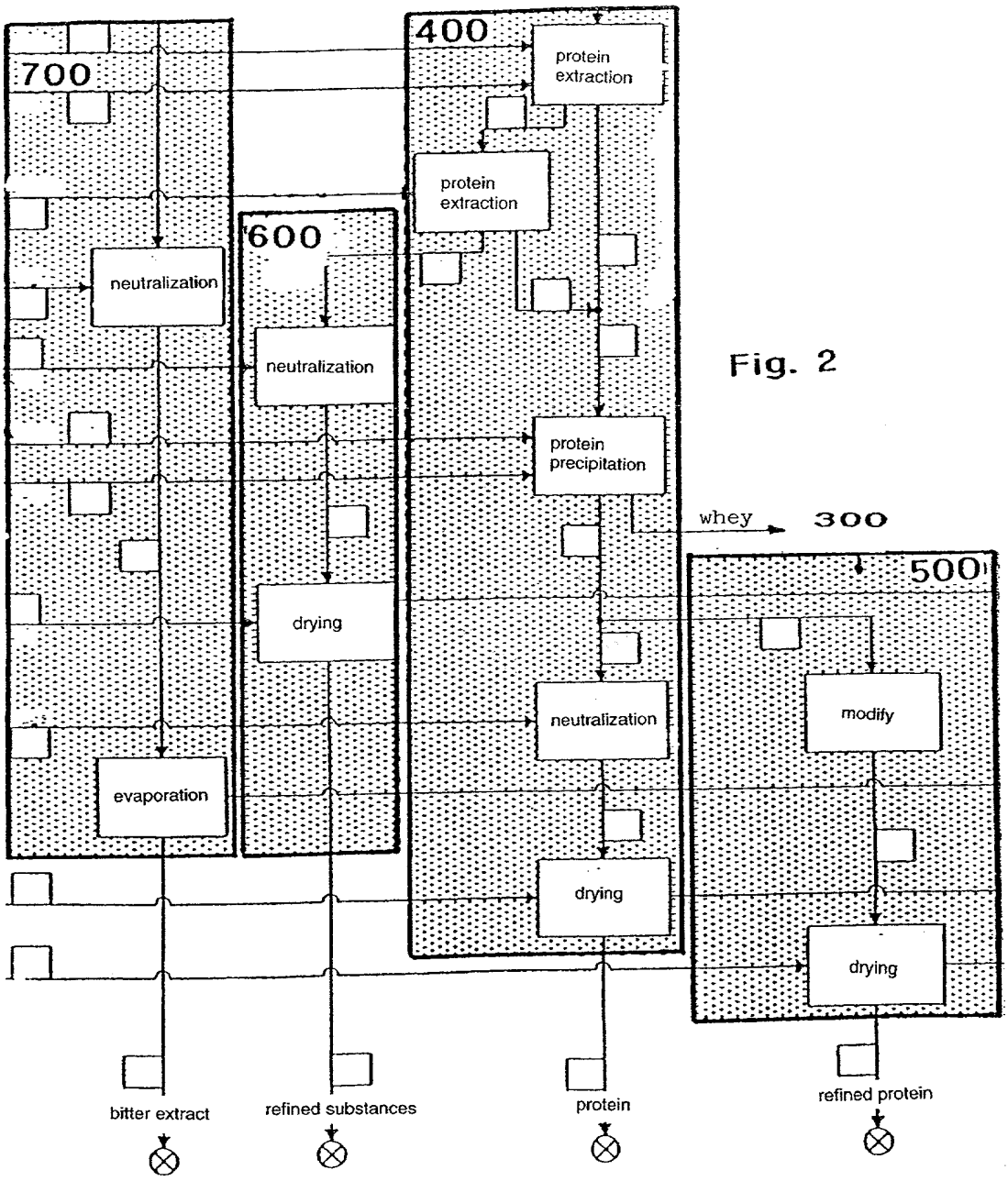

Fig. 2

Attorney's Docket No.: 785.40641X00

SUPPLEMENTAL DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As a below named inventor, I hereby declare that: my residence, post office address and country of citizenship are as stated below, next to my name; I believe I am the original, first, and sole inventor (if only one name is listed below) or an original, first, and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

METHOD FOR TREATING AND PROCESSING LUPINE SEEDS CONTAINING ALKALOID, OIL AND PROTEIN

the specification of which

___ is attached hereto.

_X_ was filed on September 17, 2001 as
United States Application Number 09/936,696
or PCT International Application Number PCT/EP00/02069
and was amended on _____.
(if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claim(s), as amended by any amendment referred to above. I acknowledge the duty to disclose all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d), of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)			Priority Claimed	
199 12 037.4 (Number)	Germany (Country)	17/03/1999 (Day/Month/Year Filed)	___ Yes	___ No
199 12 045.5 (Number)	Germany (Country)	18/03/1999 (Day/Month/Year Filed)	___ Yes	___ No

I hereby claim the benefit under title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below

(Application Number) Filing Date

(Application Number) Filing Date

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the

prior application and the national or PCT international filing date of this application:

(Application Number)	Filing Date	(Status -- patented, pending, abandoned)
(Application Number)	Filing Date	(Status -- patented, pending, abandoned)

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Title 37, Code of Federal Regulations, Section 1.56
Duty to Disclose Information Material to Patentability

(a) A patent by its very nature is affected with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclosure information exists with respect to each pending claim until the claim is cancelled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of a claim that is cancelled or withdrawn from consideration need not be submitted if the information is not material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclosure all information known to be material to patentability is deemed to be satisfied if all information known to be material to patentability of any claim issued in a patent was cited by the Office or submitted to the Office in the manner prescribed by ¤¤1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:

(1) Prior art cited in search reports of a foreign patent office in a counterpart application, and

(2) The closest information over which individuals associated with the filing or prosecution of a patent application believe any pending claim patentably defines, to make sure that any material information contained therein is disclosed to the Office.

(b) Under this section, information is material to patentability when it is not cumulative to information already of record or being made or record in the application, and

(1) It establishes, by itself or in combination with other information, a prima facie case of unpatentability of a claim; or

(2) It refutes, or is inconsistent with, a position the applicant takes in:

(i) Opposing an argument of unpatentability relied on by the Office, or

(ii) Asserting an argument of patentability.

A prima facie case of unpatentability is established when the information compels a conclusion that a claim is unpatentable under the preponderance of evidence, burden-of-proof standard, giving each term in the claim its broadest reasonable construction consistent with the specification, and before any consideration is given to evidence which may be submitted in an attempt to establish a contrary conclusion of patentability.

(c) Individuals associated with the filing or prosecution of a patent application within the meaning of this section are:

(1) Each inventor named in the application;

(2) Each attorney or agent who prepares or prosecutes the application; and

(3) Every other person who is substantively involved in the preparation or prosecution of the application and who is associated with the inventor, with the assignee or with anyone to whom there is an obligation to assign the application.

(d) Individuals other than the attorney, agent or inventor may comply with this section by disclosing information to the attorney, agent, or inventor.